# Organic Extractables in Primary-Treated Municipal Wastewater and Uptake in Exposed Juvenile Chinook Salmon. A Preliminary Study at Iona Island Sewage Treatment Plant, Vancouver, B.C.

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by

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# PREFACE

The work described herein formed half of an interdisciplinary study conducted in the summer of 1982 by scientists at the West Vancouver Laboratory of Fisheries and Oceans, Canada. The objective was to gather information on the chemical composition of municipal wastewater from the Iona Island Sewage Treatment Plant and to study its effects on exposed juvenile chinook salmon. This knowledge was intended for use by the Greater Vancouver Sewage and Drainage District - the operators of the plant - and by federal and provincial regulatory agencies. Prior to the commencement of the study the plant operator had been served with a court order to submit a report detailing how, and at what public expense, the environmental problems associated with the existing outfall could be corrected.

When the experiment commenced there was at first a degree of overlap between the necessary methods development work and the analysis of real samples. This, together with the short time-frame, resulted in the volume of quantitative data available on wastewater composition being quite limited. The work was planned to identify and measure the most abundant organics present rather than to focus on measuring selected toxicants. This tended to complicate the quantitation aspects of the analytical work because of the wide structural variety of target compounds.

The fish exposure test was terminated abruptly after three weeks, following a massive infestation of the test animals with a naturally occurring parasite and the failure of the dilution water pump. Although this complicated the interpretation of the bioaccumulation data, it was considered that the 1982 work should be published as a technical report. The value of this work lies in the guidance it gave for improved experimental design in the 1983 study. Moreover, the information on the qualitative composition of the sewage and the uptake work were of interest because of the general lack of detailed study of either aspect in the Fraser River estuary. The data reported here are examined and compared to the work of other investigators. The results of the more detailed study conducted in 1983 will be reported elsewhere.

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## ABSTRACT

Rogers, I. H., I. K. Birtwell, and G. M. Kruzynski. 1986. Organic extractables in primary-treated municipal wastewater and uptake in exposed juvenile chinook salmon. A preliminary study at Iona Island sewage treatment plant, Vancouver, B.C. Can. Tech. Rep. Fish. Aquat. Sci. 1433: 35 p.

Primary-treated municipal wastewater samples from the Iona Island Sewage Treatment Plant, Vancouver, British Columbia were analysed by gas chromatography with mass spectrometric detection to determine the qualitative composition of the extractable organics. An analytical method was developed for quantitation of organics in wastewater samples spiked with internal standards. The major extractable compounds in several sewage samples were quantified by means of gas chromatography with flame ionization detection. Recorded mass spectra showed the presence of fatty acids, petroleum hydrocarbons, aromatic acids, caffeine, nicotine, fecal sterols and a number of polynuclear aromatic hydrocarbons (PAH's) of low molecular weight. Few chlorinated organics were detected. However, the presence of a number of organics toxic to aquatic life was recorded.

Juvenile chinook salmon (Oncorhynchus tshawytscha), were exposed experimentally to 0, 1, 6 and 16% concentrations of Iona Island sewage in dilution water pumped from the Fraser River North Arm. Fish were sampled at three to five day intervals and pooled carcasses and livers analysed for the uptake of toxic organics. However, the exposure study was terminated prematurely due to high mortalities induced by a pathogenic parasite infection, together with plant problems. Thus the fish may not have been in a healthy condition during part of the exposure and the uptake data must be interpreted with caution.

Analysis of fish carcass extracts by electron capture gas chromatography showed the uptake of tetrachlorophenol, pentachlorophenol, DDE, DDD, DDT, PCB's and a series of five unknowns which did not match any common pesticides or herbicides. The uptake of $\Sigma$ DDT, PCB's and chlorophenols appeared to increase in proportion to the level of exposure. However, only in the case of tetrachlorophenol was uptake statistically related to degree of exposure. Higher levels of tetrachlorophenol were measured in carcasses of salmon exposed to 6% sewage than in those exposed to 16% concentrations. A possible explanation for this finding was suggested. The PCB's present in fish carcasses showed a degraded pattern intermediate between that of Arochlor 1254 and 1260.

The study was repeated in June - August 1983 with precautions taken to protect the fish from waterborne pathogenic diseases and with improvements in experimental design and analytical methodology.

## RESUME

Rogers, I. H., I. K. Birtwell, and G. M. Kruzynski. 1986. Organic extractables in primary-treated municipal wastewater and uptake in exposed juvenile chinook salmon. A preliminary study at Iona Island sewage treatment plant, Vancouver, B.C. Can. Tech. Rep. Fish. Aquat. Sci. 1433: 35 p.

Des échantillons d'eau ayant subi un traitement primaire à l'usine municipale de traitement des eaux usées d'Iona Island de Vancouver (C.-B.) ont été analysés par chromatographie gazeuse et détection par spectrométrie de masse afin d'en déterminer la composition qualitative des substances organiques extractibles. Les auteurs ont mis au point une méthode analytique pour la mesure quantitative des produits organiques présents dans des échantillons d'eaux usées auxquels on avait ajouté des substances témoins. Les plus importants composés extractibles de plusieurs échantillons ont été dosés par chromatographie gazeuse et détection par ionisation de flamme. Les spectres de masse indiquaient la présence d'acides gras, d'hydrocarbures de pétrole, d'acides aromatiques, de caféine, de nicotine, de stérols fécaux et de plusieurs hydrocarbures aromatiques polynucléaires de faible masse moléculaire. Peu de composés organiques chlorés ont été décelés, mais bon nombre de substances organiques toxiques pour la vie aquatique l'ont été.

Des saumons quinnat juvéniles (Oncorhynchus tshawytscha) ont été exposés à des concentrations de 0, 1, 6 et 16% d'eaux usées d'Iona Island diluées par de l'eau pompée du bras nord du Fraser. Des poissons étaient sacrifiés à intervalles de trois à cinq jours et leurs carcasses et foies étaient regroupés pour analyse des produits toxiques absorbés. Des taux de mortalité élevés dus à une infection par un parasite pathogène et des problèmes d'exploitation à l'usine on cependant mis fin prématurément à cet essai. Il est possible que les poissons n'aient pas été en bon état de santé au cours d'une partie de l'exposition et les données d'assimilation doivent donc être interprétées avec prudence.

L'analyse des extraits de carcasses par chromatographie gazeuse et capture d'électrons a montré qu'il y avait eu assimilation de tétrachlorophénol, de pentachlorophénol, de DDE, de DDD, de DDT, de BPC et de cinq autres substances que l'on n'a pu apparier à des pesticides ou herbicides courants. L'assimilation des Σ DDT, BPC et chlorophénols semblait augmenter en fonction du niveau d'exposition, mais une corrélation statistiquement significative n'a été notée que pour le tétrachlorophénol. Des teneurs plus élevées en ce produit ont cependant été notées chez les poissons exposés à la concentration d'eaux usées de 6% plutôt qu'à celle de 16%. Une hypothèse a été formulée pour expliquer ce phénomène. Les BPC présents dans les carcasses se présentaient sous une forme se situant entre celles des Arochlor 1254 et 1260.

L'étude a été répétée de juin à août 1983 et on a pris les précautions nécessaires pour protéger les poissons des agents pathogènes présents dans l'eau. La conception expérimentale et la méthodologie analytique ont aussi été améliorées.

# INTRODUCTION

Two events in the past fifteen years have led to a dramatic increase in the generation of new information concerning the levels of organic microcontaminants in environmental samples. The first was the introduction and marketing of interfaced gas chromatographic-mass spectrometric-data processing (GC-MS) instruments that permit the detection of nanogram or sub-nanogram quantities of many organic micropollutants while recording structurally descriptive information. Very commonly these microcontaminants are present in complex matrices, which may be resolved, following cleanup, into discrete components using the great resolving capability of fused silica capillary gas chromatographic columns. The second event was the EPA Consent Decree of June 7, 1978 in the United States, wherein the Environmental Protection Agency agreed in a court settlement to establish environmental guidelines for a list of priority pollutants for which levels must be reported in all U.S. industrial wastewater discharges. The reasoning involved in the selection of the 129 compounds has been discussed (Keith and Telliard 1979). It was based on a combination of frequency of occurrence, presence of known toxic or carcinogenic effects, and the need to select compounds representing a broad range of chemical classes with diverse physical properties. In formulating policies to comply with the Consent Decree, EPA established twenty-one industrial categories for which specific guidelines were developed. Publicly owned sewage treatment plants were considered as a separate industrial category and are regulated as such.

Discharges of municipal wastewaters from the Greater Vancouver Sewerage and Drainage District (G.V.S. and D.D.) to the Fraser River estuary and nearby marine waters are a matter of concern to managers responsible for the safeguarding of important commercial fish stocks, which utilize this environment. Currently the populations of some important species - especially chinook salmon (Oncorhynchus tshawytscha) - are in decline. Water quality impairment, due to releases of municipal wastes with their B.O.D., heavy metal, and possible toxic organic loadings, may be contributory to such declines. At present only fragmentary local information is available (Garrett 1980, Cain et al. 1980, Anon. 1980b) on the nature and quantity of organics discharged, where these compounds originate, and to what degree they are toxic to aquatic life.

Of the four primary sewage treatment plants operated by the G.V.S. and D.D., the largest is located on Iona Island, at the mouth of the North Arm of the Fraser River and adjacent to Vancouver International Airport (Figure 1). This services a population of 640,000 and the incoming flow contains about 7% by volume of industrial wastes. Because stormwater also enters the mains, flows vary widely (in 1982 the daily mean flow was 0.467 and the maximum flow was 1.5 x 106 $m^3 \cdot d^{-1}$) depending on rainfall conditions. In addition, tanker trucks discharge annually about 55,000 m3 of high B.O.D. wastes, such as septic tank sludges and slaughterhouse wastes, etc. During June-July, 1982, these wastes entered the incoming sewage in concentrated slugs at the head of the system. Since then, additional storage capacity has been added and such wastes are now more gradually metered into the incoming sewage.

During the summer months, May through September, the treated sewage is disinfected by chlorination to an average chlorine residual content of 1.1 mg·L-1 before discharge. This measure, intended to protect public health, has the potential to increase the impact on aquatic life through the formation of toxic chlorinated organic compounds. The treated wastewater is held in a lagoon with a variable but low (<2-hour) retention time, cascades over a weir and into a trench in the marine intertidal foreshore before joining the waters of the Strait of Georgia. Field studies conducted in the area of the discharge have documented large fish kills correlated with low dissolved oxygen in the waters and the activities of predatory birds (Birtwell et al. 1984). Some preliminary studies, dealing with the environmental impact of the plant, have been published (Birtwell et al. 1983). These studies indicated that habitat areas within about 1 km of the discharge point were severely degraded and not used by juvenile salmon. Seawater in this area was generally depleted in oxygen due to the B.O.D. demand of organics in the sludge beds. Planning and engineering studies have been completed to change the discharge location. It is proposed that the wastes from the sewage treatment plant be discharged through diffusers located at average depths of 80 and 100 meters respectively, below the surface of the Strait of Georgia and the new works are to be completed before December 31, 1987.

We describe herein the results of trace organic analyses of sewage samples collected from the Iona Island plant during the spring and summer of 1982. We also report on analyses of juvenile chinook salmon exposed to 0%, 1%, 6% and 16% concentrations of sewage during June-July 1982. An outbreak of the protozoan parasite Ceratomyxa shasta and the failure of the diluent water pump forced termination of the salmon exposure experiment after 24 days. Because of the unknown influence of the disease on contaminant uptake and depuration, the quantitative results of fish tissue analyses should be interpreted with caution.

In June-August 1983, a modified and expanded version of the experiment was successfully completed and will be described elsewhere.

## METHODS

### Materials and Supplies

Burdick and Jackson pesticide grade solvents were used as received. Solvent blank checks were run periodically. Anhydrous sodium sulphate (certified ACS grade) was fired in a muffle furnace overnight at 600 oC. A blank extract was prepared by elution of a column packed with sodium sulphate using methylene chloride, as when extracting fish tissue samples. Florisil PR (60-100 mesh) was heated at 600°C overnight and stored in an oven at 130 oC until needed. It was then used without deactivation. Silicic acid (Mallinckrodt Chemical, 100 mesh) was heated at 500°C for eight hours and stored in an oven at 130 oC until required. It was also used without deactivation. The blank extract was passed through the Florisil and silicic

acid cleanup stages and eluates were analysed after each stage. Standard solutions of the chlorinated hydrocarbon pesticides, methoxychlor, mirex, lindane, heptachlor, heptachlor epoxide, endrin, dieldrin, chlordane, hexachlorobenzene, α- and γ BHC, α- and β-endosulfan, o-p' and p-p' DDE, o-p' and p-p' DDD, o-p' and p-p'-DDT and PCB's (Arochlor 1242, 1254 and 1260) were purchased from Supelco.

Laboratory glassware was washed with Ivory liquid soap (Proctor and Gamble Co.), rinsed well with water and baked at 325°C overnight. Glassware was hexane rinsed prior to use.

Fish Exposure Methodology

Complete details of the exposure system, ancillary water and effluent quality measurements and biological results are reported separately (Kruzynski et al. 1986 in prep.) so only a brief description is provided here.

Underyearling chinook salmon of the Qualicum River (Vancouver Island) stock were exposed to continuous flow dilutions (0, 1, 6, 16%) of Iona Island S.T.P. effluent from June 18-July 12, 1982. A total of 5400 fish of initial weight ~5 g was split equally between four fiberglass troughs (450 L). Each trough received a continuous flow of ~60 $L \cdot min^{-1}$ of diluted effluent for a 99% replacement of 0.9 hr (Sprague 1969). Ranges of temperature, dissolved oxygen, conductivity and pH were 14.0-16.5°C; 6.9-10.5 $mg \cdot L^{-1}$, 50-178 $\mu$ mho$\cdot cm^{-1}$ and 6.3-7.9 respectively. Effluent dilutions were maintained by mixing Fraser River water with effluent pumped continuously from the downstream end of a primary sedimentation pond.

Diluent water was provided via a submersible pump suspended from a log boom anchored in the North Arm of the Fraser River, adjacent to the Iona plant. The pump intake was located at a depth of about one meter and at zero tide was 2.5 to 3.0 m above the river bed. Water was pumped continuously through a 240 m length of rubber hose into two 144L fibreglass tanks where constant head was maintained by means of a PVC standpipe. Glass metering tubes, fitted into laboratory rubber stoppers, were used to adjust the rates of delivery of diluent water into the exposure troughs.

Effluent was delivered through PVC pipes into a third constant head tank at a rate of 70 L$\cdot$min-1 by means of two magnetic drive pumps. Initially intake depth varied from ~5-30 cm with fluctuations in pond level. Subsequent repositioning of the pumps on a styrofoam float ensured a fixed intake depth of ~30 cm relative to the surface regardless of level fluctuations. Pump materials in contact with effluent were glass-filled polypropylene (housing), Buna "N" rubber O-rings, ceramic magnet and stainless steel.

The header tanks and exposure troughs had previously been used for several years in laboratory fish culture work so that this potential source of trace contaminant leaching was considered to be minimal. However, despite efforts to reduce the chances of contamination from the pumping system, subsequent analyses indicated an elevation with time of zinc and copper in

fish exposed to Fraser River water. Controlled leaching tests conducted subsequently indicated that the 240 m hose was likely the source of this heavy metal contamination. Details are reported in Kruzynski, Birtwell, and Rogers 1986 in prep.

Municipal Wastewater Samples

Grab samples (4 L) were collected by G.V.S. and D.D. plant personnel in prewashed glass bottles from the surface of the chlorine contact tank near the outfall weir at Iona Island Sewage Treatment Plant on April 26, May 14, August 19 and September 15, 1982. Composite samples (24 h) were collected near the same location on March 16 and 25, April 21, 22 and 26, June 13 and September 15, 1982. These were taken with a flow-proportioned Sirco automatic sampling device used for routine wastewater monitoring in which the sample is maintained at 4°C during collection. All wastewater samples were picked up from the plant by West Vancouver Laboratory personnel on the morning of the day the samples were taken and analysis commenced the same day in West Vancouver.

During the fish exposure study, we attempted to collect a continuous sample of effluent by means of a siphon (Intramedic medical formulation polyethylene tubing) dripping into a glass container packed in dry ice. Because of frequent clogging overnight, the planned collection volume of 1.0 to 1.5 $L \cdot day^{-1}$ was not regularly acheived. However, daily attendance ensured that effluent was sampled every day. The frozen effluent "buttons" were kept in a freezer prior to processing. In the laboratory these samples were thawed and blended to yield two composite samples covering the time intervals of June 18-25 and July 3 - July 10, 1982. The sample for the intervening period June 25 - July 3 was lost due to container breakage at the plant. Of the 3-4 L of sample collected during each interval, 1200 ml was subsampled and subsequently extracted.

Extraction and Fractionation Of Wastewater Samples

The possibility of extracting sewage samples with either diethyl ether or methylene chloride in separatory funnels to remove base/neutral fractions was investigated. Emulsification problems dictated that a different approach be pursued.

Further tests were run using a liquid/liquid extractor designed for solvents more dense than water with a sample volume of 1500 ml. Optimum recoveries were achieved using methylene chloride as extracting solvent for the base/neutrals. After 16 hours continuous extraction at pH 10, the extract was removed, sample pH adjusted to 2 and extraction recommenced with methylene chloride/diethyl ether (4 to 1) for 16 hours. The extract was drawn off and the sample transferred to a separatory funnel for extraction with diethyl ether. Two ether extracts were collected and combined with the methylene chloride/diethyl ether extract for recovery. This method avoided emulsion formation and the development work has been described (Rogers et al 1983).

A mixture of equal parts of 1,2-dibromo-4,5-dimethoxybenzene and methyl margarate was selected as an internal standard. A solution in methylene chloride was spiked into base/neutral and acid extracts prior to evaporation. The spiked concentration of the internal standards in the wastewater samples was 200 $\mu g \cdot L^{-1}$. Acid fractions were methylated using an ethereal solution of diazomethane. All extracts were concentrated to 1 mL in preparation for analysis.

Experiments were conducted in which solutions of mixtures of 10 base/neutrals and 3 acids, together with the two internal standards were spiked into water and recovered by the liquid/liquid extraction procedure. The compounds were selected because of their known occurence in the wastewater (GC-MS analysis, see later) and because they covered the range of volatility of the extractable organic components present in the base/neutral and acid fractions as indicated by retention times, determined by gas chromatography. Evaporation of diluted solutions of the spiking standard mixtures was performed using both a rotary evaporator and a Kuderna - Danish apparatus. Analysis of the recovered concentrates by gas chromatography did not show measurable differences between the two procedures so the rotary evaporator was used throughout the study.

Analysis of Extracts by Gas Chromatography - Mass Spectrometry (GC-MS) and Gas Chromatography (GC-FID)

Extracts were analyzed qualitatively using a Hewlett-Packard 5992 B GC-MS desktop instrument fitted with a capillary column injection system and cryogenic cooling. The oven temperature was programmed from 80°C to 290°C at 10°C $min^{-1}$. The ion source was set at 70 electron volts and the instrument operated in the peakfinder mode using perfluorotributylamine for automatic internal calibration. A 30 m x 0.32 mm Durabond - 5 fused silica capillary column was used in the splitless mode. Helium was the carrier gas at a flow rate of 1.5 $mL \cdot min^{-1}$. Mass spectra were manually identified with the aid of the Eight Peak Index (Aldermaston file) and the EPA - NIH file. All major components identified were confirmed by comparison of mass spectra and retention times with those of standards.

Quantification of major extractable components of sewage was accomplished by gas chromatography using a flame ionization detector (GC-FID). The instrument was a Hewlett-Packard 5700 and contained a capillary column similar to that used in the GC-MS analysis and operated under equivalent conditions. Ten base/neutral components (compounds 1 to 10, Table 1) and three acidic components of the wastewater (compounds 11-13, Table 1) were selected as quantification standards. The retention time and response factor of each compound relative to the two internal standards was determined and entered into the memory of a Hewlett-Packard 79850 A liquid chromatography terminal, used as an integrator. After checking the procedure against various dilutions of the mixture of thirteen standards (including the two internal standards) in water, the concentrations of all major chromatographable components in six samples of Iona Island wastewater were determined. The compounds present and their ranges are listed in Table 2 (base/neutrals) and Table 3 (acids).

Extraction of Tissue Samples from Exposed Chinook Salmon

Carcasses of chinook salmon (whole animals minus livers, guts and brains) were allowed to thaw and the excess water decanted. Twenty fish per sample were weighed out from the first sampling period (17th June, weights ranged from 68.5 g to 94.3 g). Thereafter ten fish were used per sampling period (range 37.0 g to 63.2 g) from the population exposed to 1%, 6% and 16% sewage and from the control. Each sample was mixed with four times it's weight of anhydrous sodium sulphate using a clean spatula and the mixture was poured on to aluminum foil, prewashed with methylene chloride. The labelled packages were kept in a refrigerator for 16 hours to allow water and lipids to soak into the dessicant. The sample was then finely ground using first a clean mortar and pestle followed by disintegration in a Sorvall Omni-Mixer. A glass chromatographic column with Teflon stopcock was plugged with cotton wool and covered with a layer of sodium sulphate. The column walls were washed down with methylene chloride (40 mL), which was allowed to run to waste. The homogenized sample was then poured into the column, gently compacted, and covered with a bed of anhydrous sodium sulphate. The stainless steel beaker from the Sorvall mixer and the mortar and pestle were rinsed twice with methylene chloride and the washings added to the extraction column. Methylene chloride was allowed to percolate through the sample until it reached the stopcock. The latter was closed and the solvent left in contact with the sample for an hour. The extract was collected (300 mL) and a further volume (50 mL) taken separately and evaporated to check the completeness of lipid removal. Further volumes of extracting solvent were collected as needed and the total extract evaporated using a rotary evaporator. The extracts were transferred to tared vials and allowed to evaporate to constant weight in a fumehood. The lipid yields were determined gravimetrically.

Cleanup and Analysis of Fish Extracts

Aliquots of lipid extracts (500 mg) were separated by gel permeation chromatography (GPC) on Bio-Beads S-X3 (200-400 mesh) according to an established method (Tindle and Stalling 1972). The lipid sample, dissolved in methylene chloride, was injected on to the bottom of a column, 48 x 2.5 cm, packed with 60 g Bio-Beads S-X3. The eluting solvent was a mixture of equal parts cyclohexane and methylene chloride at a flow rate of 5 $mL \cdot min^{-1}$. The first 150 mL eluate contained lipids, fatty acids, aliphatic hydrocarbons and some phthalate esters and was discarded. The micropollutants were collected in the next 170 mL of eluate.

For purposes of quality assurance, 1 g samples of corn oil, free of PCB's and DDT-related compounds, were spiked at the level 200 $ng \cdot g^{-1}$ with standard solutions of TCP, PCP, DDE, DDD, DDT and PCB's (Arochlor 1254). The samples were processed through the same stages of cleanup as the fish extracts to determine recovery values on the individual standard substances. Blank extracts were prepared from unspiked corn oil and procedural blanks were also prepared by taking the residue from sodium sulphate blanks through each step of the cleanup procedure.

After the GPC cleanup, the samples were methylated with diazomethane to convert the chlorophenols to their anisole derivatives and prevent losses during column chromatography. The extracts were refined by passage through a small glass chromatographic column (15 x 1.5 cm), dry packed with Florisil as described by earlier workers (Mills 1959; Johnson 1962). The column was prewashed with petroleum ether (40 mL, b 30 - 60 °C) and the sample transferred to the bed surface. The contaminants were eluted with diethyl ether/petroleum ether (6:94; 200 mL). This fraction contained the PCB's and most chlorinated pesticides. A second eluate was collected using diethyl ether/petroleum ether (40:60;200 mL). This fraction was examined for the possible presence of some more polar pesticides including endrin, dieldrin, diazinone, methyl and ethyl parathion and α and β-endosulfan (Anon 1980a).

The first Florisil fraction was chromatographed on a bed of silicic acid to separate PCB's from chlorinated hydrocarbon pesticides according to an established method (Armour and Burke 1970). A slurry of activated silicic acid (5g) in hexane (80 mL) was poured into a small glass chromatographic column (100 x 22 mm) plugged with cotton wool. The hexane was drained down to the bed surface and discarded. The sample was added to the bed surface as a concentrated solution in hexane and the column eluted with hexane (50 mL) containing 0.5% benzene to recover the PCB fraction. The pesticide fraction was then removed with diethyl ether/hexane (1:4;150 mL). All fractions were taken just to dryness in a rotary evaporator. The residues were then dissolved in exactly 1 mL hexane prior to analysis.

Fish extracts and spikes recovered from corn oil were analysed by electron capture gas chromatography (GC-EC) using a Hewlett-Packard 5711 gas chromatograph fitted with a Ni63 electron capture detector. PCB's, DDT, DDD, DDE, tetra- and pentachlorophenol (TCP and PCP) were measured using external standard mixtures of known concentrations, which were adjusted as required to give linearity with the samples. The capillary column and operating conditions were as described for analysis of sewage samples.

## RESULTS AND DISCUSSION

### Organic Contaminants in Wastewater

The results of spike recovery trials from water on mixtures of base/neutral and acidic components are listed in Table 1. The compounds selected were representative of the classes of organics detected in Iona Island sewage extracts and covered a wide range of volatility, as evidenced by retention time (R.T.) on a Durabond-5 GC column. Although phenol, m-cresol and 2,4-xylenol are weakly acidic compounds, they are recovered mainly with the base/neutral fraction under our conditions of extraction. Had the pH of the spiked water samples been raised to levels higher than 10 prior to extraction, it is anticipated that these compounds would not have been extracted with the base/neutrals but would have remained in the acidic fraction.

The recoveries of the spiking standards (Table 1) were greater than 80% in all cases except for phenol, butylbenzene and nicotine. Caffeine gave a high recovery (153%). It is generally considered that recoveries of micropollutants >80% are acceptable (see for example Thompson et al. 1977; Bickford et al. 1980). The difficulty in recovering phenol by extraction of dilute aqueous solutions is well known. Work in support of methods development for the Consent Decree Priority Pollutants in the United States underlined this. Using a batch liquid/liquid extraction technique with methylene chloride as solvent, recoveries of 48 ± 1% were measured for spiked water samples and 42 ± 1% for spiked samples of municipal effluent (Bickford et al. 1980). The difficulties experienced in obtaining quantitative data with nicotine and caffeine may be due to the polar nature of these compounds. The peaks recorded for caffeine on GC charts were relatively wide, indicating a strong interaction with the column liquid phase. Probably choice of a more polar column than Durabond-5 would improve the quantitation of this component. However, this might also result in less acceptable results for the other, less polar substances.

As internal standards for wastewater analysis we chose 1,2-dibromo-4,5-dimethoxybenzene and margaric acid. Brominated compounds have not been found in Iona Island Sewage Treatment Plant wastewater samples and the compound selected gave a readily identifiable mass spectrum and was not subject to interferences. Margaric acid is a minor component in the wastewater samples so there is some inaccuracy introduced by this choice. On the other hand it structurally resembles the fatty acids which are the main components of the acidic fraction of the wastewater. Both internal standards were spiked into the base/neutral fraction and into the acidic fraction so that they could be used independently, if necessary, to verify data on the composition of sewage samples.

The base/neutral substances identified qualitatively by GC-MS analysis of eleven sewage samples are listed in Table 2 in order of increasing retention time. Many additional spectra were recorded but could not be identified because of interferences or lack of fragments with mass to charge ratios greater than about 140. Many of these spectra closely resemble the mass spectra of aliphatic hydrocarbons found in waste petroleum. No single sewage sample contained all the compounds listed in Table 2. Because retention times varied from day to day, depending on gas flow rates and oven temperatures, the order of the compounds in Table 2 was derived by calculating retention times relative to the internal standard 1,2-dibromo-4,5-dimethoxybenzene. It was then possible to combine the identifications from several sewage samples. The only chlorinated substances identified in sewage samples were trichloropropene, two isomers of dichlorobenzene and two monochloro- compounds with molecular weights 218 and 302. The mass spectrum of the former closely matched the published mass spectrum of 2-benzyl-4-chlorophenol. The GC-FID chart corresponding to a base/neutral extract of an Iona Island sewage sample is reproduced in Figure 2. Since the same GC column is used in the GC-FID analysis as in the GC-MS determination, the compounds identities can be transferred to the corresponding peaks on the GC-FID charts. Figure 2 illustrates the complexity of the organic fraction in the municipal wastewater sample and, by inference, the number of components not identified by GC-MS anlysis. The presence of some free fatty acids in the base/neutrals was noted.

In the same fashion the compounds identified in the acid fraction of the ten sewage samples are listed in Table 3. The prevailing acids were normal chain fatty acids most probably derived from household cooking residues. A series of aromatic acids was measured and a few dicarboxylic acids were also present. No chlorinated substances were identified by GC-MS analysis in the acid fractions. The GC-FID chart of the acid fraction of the same sewage sample shown in Figure 2 is reproduced in Figure 3. It was observed that some base/neutral compounds - especially paraffin hydrocarbons - are difficult to remove completely in the base/neutral extraction. Traces of these residues, including cholesterol and coprostanol, appear in the acid fractions. Similar difficulty was encountered by workers in the Southern California Coastal Research Project when analysing sewage samples from the Los Angeles County wastewater treatment plant (Gossett et al. 1983a)

Using response factors measured on the spiking standards listed in Table 1, the memory of the GC-FID integrator was calibrated to allow concentrations of all major extractable organic wastewater components (based on peak height) to be quantified. Response factors were assumed for other compounds, not present in Table 1, using the same value as that for the spiking standard with the closest matching retention time. The data are shown as ranges for six wastewater samples analysed in Tables 2 and 3. The most plentiful components measured were several normal fatty acids. These ranged in concentration from 0.2 to 1.8 $mg \cdot L^{-1}$. The same compounds were present in Los Angeles wastewater (Gossett et al. 1983a) in the 1.0 to 2.5 $mg \cdot L^{-1}$ range. Aromatic acids (Benzoic, phenylacetic and phenylpropionic) were also prominent in Iona Island wastewater (range 7 to 344 $\mu g \cdot L^{-1}$). Only benzoic acid (400 $\mu g \cdot L^{-1}$) was reported in Los Angeles sewage. No chlorinated acidic compounds were present in sufficiently high concentration to be detected by the analytical methods used in the present study. In the Los Angeles work, GC-EC was used and also selected ion monitoring (GC-MS-SIM) for the EPA priority pollutants. This technique is more sensitive than the normal full scan GC-MS by a factor of 100 to 1000. Thus 2,4,5- and 2,4,6-trichlorophenol, tetra- and pentachlorophenol were reported in the Los Angeles wastewater in the range 0.12 to 2.3 $\mu g \cdot L^{-1}$.

The most plentiful base/neutral components in the Iona Island wastewater fell in a concentration range about an order of magnitude below that of the major acidic components. The most abundant compound was $\alpha$-terpineol, used as a germicide in household disinfectants (range 21 - 191 $\mu g \cdot L^{-1}$). Other relatively common neutral substances were normal paraffins from petroleum ($C_{12}$ to $C_{28}$), phthalate esters, caffeine, some cresols and xylenols - also probably used as household disinfectants - and some alkylbenzenes, probably arising from waste fuel oils. The Los Angeles list of base/neutral substances was similar except that $\alpha$-terpineol was not identified and a number of water-miscible organic solvents, were reported. Fifteen pesticides, PCB's and other organochlorine compounds were present in the Los Angeles wastes in the range 0.94 to 0.003 $\mu g \cdot L^{-1}$ but were undetected in the Iona Island wastewater samples.

Some previous workers have analyzed isolated samples of Iona Island Sewage Treatment Plant wastewater. In a study of the levels of organic contaminants in wastewaters and stormwaters from the Vancouver area, Koch et

al (1977) reported total hydrocarbons in the range 6.1 - 15.8 $mg \cdot L^{-1}$ in the influent to the plant. The results were calculated as isooctane. Total phenols were measured as 12.5 $\mu g \cdot L^{-1}$ and surfactants as 0.46 - 0.53 $mg \cdot L^{-1}$ (methylene blue active substances MBA's). Cain et al. (1980) reported data on 24-hour composite samples of wastewater collected on March 8, 1978. They found fatty acids (to 200 $\mu g \cdot L^{-1}$ stearic acid), phthalate esters (to 50 $\mu g \cdot L^{-1}$ dimethyl isophthalate), caffeine, pentachlorophenol (6 $\mu g \cdot L^{-1}$), tetrachlorophenol (30 $\mu g \cdot L^{-1}$), hexachlorobenzene (0.004 $\mu g \cdot L^{-1}$) and PCB's (0.3 $\mu g \cdot L^{-1}$). Three samples collected in November 1979 were analysed by Radian Inc. of Austin, Texas under contract to Environment Canada (Anon 1980b). 2,4-Dimethylphenol, 2-chlorophenol and 2-nitrophenol were identified by GC-FID at concentrations too low to permit confirmation via GC-MS analysis.

The compounds identified in Tables 2 and 3 show a strong resemblance to the list of substances identified by Garrison et al 1976, working at the Southeastern Water Research Laboratory of the Environmental Protection Agency in Athens, Georgia. They analyzed wastewater samples supplied to them by the Advanced Waste Treatment Laboratory of their agency, located in Cincinnati. The latter facility operates pilot-scale treatment plants under well documented and controlled conditions. Garrison et al. did not report finding normal paraffins in domestic sewage and the alkylbenzenes, alkylnaphthalenes and phenols seen in the current Iona Island samples were either absent or less prominent in the Cincinnati raw sewage. This may be related to the fact that an oil refinery is on line to Iona Island STP. Alternatively the data may reflect the uncontrolled input of waste oil and fuel from service stations, street runoff and miscellaneous spills of petroleum products.

Other research workers have studied sewage treatment plants in Seattle and Southern California as sources of petroleum wastes entering the oceans. Thus the Westpoint primary sewage effluent discharged to Puget Sound from the city of Seattle, Washington has been estimated to convey 475 metric tons per year of aliphatic hydrocarbons and approximately 1 metric ton per year of three to seven-ring PAH's (Barrick 1982). It has also been estimated that the major discharges of municipal wastewaters in Southern California carry about $17.4 \times 10^3$ metric tons total hydrocarbons annually to the ocean or 4.9 g per capita day (Eganhouse and Kaplan 1982). Recently the composition of the water soluble fractions of various fuel oils has been published (Coleman et al. 1984). The major components are naphthalene, anthracene, phenanthrene and their alkyl derivatives of which a number were detected in the base/neutral fractions of Iona Island wastewaters (Table 2).

The base/neutrals of two composite sewage samples, collected during the fish exposure period 17 June to 7 July, 1982, yielded GC-FID and GC-MS data revealing the presence of several unfamiliar contaminants. The mass spectra were consistent with a series of poly (organosiloxanes), which have widespread industrial applications. The presence of poly(organosiloxanes) in municipal sewage sludge and in sediments from the Potomac River, Washington, D.C. has been reported (Pollenbarg 1979). It is puzzling that only traces of these compounds were noted in all other wastewater samples analysed during the 1982 and 1983 studies (unpublished data). Perhaps there may have been a spill during the fish exposure period in 1982 or some portion of the sampling

apparatus may have been contaminated with these materials. However, it appears that withdrawal of wastewater samples from the surface layer of the sedimentation tank was unlikely as the intake to the sampling pump was located about 30 cm below the water surface.

Electron capture gas chromatography (GC-EC) was not applied to sewage analyses in our 1982 studies. Chlorinated organics, if present in the wastewater, were at levels below detection limits for GC-FID and GC-MS. This was apparent later when tissues of sewage-exposed fish were analyzed. Consequently sewage samples were routinely quantified for tetra- and pentachlorophenol by GC-EC during subsequent studies conducted in June-August, 1983.

No measurements were made on volatile compounds in the Iona Island wastewater during 1982. A limited study of this aspect was conducted earlier (Birtwell et al. 1983). The principal volatiles detected in receiving waters near the outfall were chloroform, trichloroethylene, tetrachloroethylene, benzene, toluene and the three isomers of xylene.

## Uptake in Exposed Fish Tissues

For purposes of quality assurance, the recovery of a suite of five chlorinated compounds, found in tissues of the exposed fish, was measured on spiked samples of corn oil at the 0.2 $\mu g \cdot g^{-1}$ level. The recoveries and standard deviations (n=10) were as follows: TCP 102% ± 16.0, PCP 107% ± 12.7, DDE 76% ±18.9, DDD 78% ± 23.9 and DDT 95% ± 18.8. It is probable that less variance would have occurred if an internal standard had been used rather than an external standard. Analysis of procedural blanks showed the absence of interferences that would affect the integration of peaks on gas chromatographic charts for the above suite of chlorinated pesticides and also for PCB's.

The results of analyses of carcasses of sewage-exposed salmon are given in Table 4. The tissues contained low levels (to 110 $ng \cdot g^{-1}$ wet tissue) of a degraded PCB mixture containing Arochlor 1254 and 1260. However, in three of the control samples, the levels of PCB's were below the limit of quantitation. This has been defined as a signal at least ten times the noise level on the GC chart (Environmental Protection Agency 1982). We measured the limit of practicable quantitation of PCB's in wet fish tissue as about 10 $ng \cdot g^{-1}$ as Arochlor 1254. Using an average lipid content of 3%, the limit of quantitation may also be stated as 300 $ng \cdot g^{-1}$ lipid. Thus some of the PCB levels were below or near the bottom end of the range of quantitation.

In this work PCB's were quantified by averaging the integrals for a series of major peaks which were clearly resolved from other components. A solution of Arochlor 1254 of known concentration was used as an external standard. Only three cleanly resolved peaks were present in the standard so the instrument was calibrated against these three components. The corresponding peaks in the GC-EC charts from fish extracts, after silicic acid cleanup, were integrated and the mean of the integrals for the three peaks

used to compute the level of PCB's as Arochlor 1254. Difficulties in quantifying PCB's because of weathering, differential metabolism or overlapping Arochlor patterns, are well known and have been recently reviewed (Reynolds 1983).

The levels of PCB's measured in the exposed salmon may be compared to earlier data on wild fish captured in the Lower Fraser River or in the marine waters near the mouth of the river. A survey was made of PCB's and chlorinated hydrocarbon pesticide levels in fish, crabs and shellfish of the Lower Fraser River and estuary collected in 1972-73 (Albright et al. 1975). PCB's were detected in muscle tissue of various fish species more frequently than the various chlorinated pesticides studied. Moreover the frequency of occurrence of PCB's and chlorinated pesticides was greatest in fish taken from stations in the metropolitan area. Adult chinook salmon contained a mean level of 86.8 ng·g-1 PCB's whereas levels were undetectable in sockeye salmon adults. Highest concentrations of PCB's were measured in squawfish (to 1894 ng·g-1). Work on analysis of various fish species from the area has been summarized (Garrett 1983). No further data were recorded for levels in salmon .

The PCB content of coho salmon (O. kisutch) muscle tissue from Lake Ontario (measured as a mixture of equal parts Arochlors 1254 and 1260) was reported for fish captured in 1976 (Norstrom et al. 1978). Mean levels were 8170 ± 4490 ng·g-1 wet weight (n=28). Contaminants were also analysed in the 1981 fall run of coho salmon in each of the Great Lakes (DeVault and Weishaar 1982). The levels were greatest in Lake Ontario where total PCB's (Arochlors 1260, 1245 and 1248) of 1370 to 3300 ng·g-1 were measured. The lowest values were reported for the Lake Superior samples which contained 100 ng·g-1 of total PCB's. Thus it is clear that the PCB levels measured in the chinook salmon exposed to Iona Island sewage were low compared to environmental values measured in more highly polluted systems.

Oregon moist pellets fed to the salmon during the exposure study were analyzed in the subsequent 1983 study and a level of 137 ng·g-1 PCB's (as Arochlor 1260, wet weight basis) was measured together with values of 42 ng·g-1 and 10.5 ng·g-1 for DDE and DDT respectively. Other workers have also analysed samples of Oregon moist pellets (Mac et al. 1979) and reported results in the range of 100 to 230 ng·g-1 for PCB's and 10 to 340 ng·g-1 for DDE. There is thus a likelihood of uptake of these contaminants from the feed as well as from the exposure to sewage. The likely source of these contaminants in Oregon moist pellets is fish oil, which is used as one of the ingredients.

Comparison of our uptake data for PCB's with data collected from the analysis of marine species near municipal sewage outfalls off Southern California requires caution, since those fish were exposed to both possible mechanisms of uptake and may have lived in the area of the outfalls for extended time periods. Nevertheless, the levels reported for Arochlor 1254 from white croaker muscle tissue from the Metropolitan Los Angeles area measured in the range 11 to 3202 ng·g-1 wet basis depending on sampling station (Gossett et al. 1983b).

The presence of TCP and PCP in sewage-exposed salmon carcasses was scarcely surprising in view of the widespread use in British Columbia of these chemical wood preservatives. The environmental problems associated with the wood preservation industry in this province have recently been reviewed (Henning and Konasewich 1984). The product used commercially comprises an 80% mixture of two isomers of TCP with 20% PCP, whereas the product retailed to the public is largely PCP. The presence of TCP in the test fish implies that a significant problem is associated with surface runoff or disposal of residual chlorophenols at commercial wood preservation operations in the collection area for the Iona Island plant.

The data recorded in Table 4 indicate the presence of both TCP and PCP in the stock of fish shortly after arrival from the hatchery (day-1). This could be due to a combination of low levels of these contaminants in the water and fish diet used at the hatchery. The levels in the control fish were relatively constant for both TCP and PCP until the last sampling period, when a significant increase was noted, presumably because of higher levels in the dilution water from the Fraser River North Arm. Levels of TCP and PCP in fish exposed to 16% sewage did not seem higher than the controls except for the final two sampling periods, when an apparent increase was observed for both compounds. The levels of TCP measured in fish exposed to 6% sewage were the highest of the three groups and significantly elevated above the controls throughout. In this case also, the highest levels were measured in fish from the final two sampling periods. The levels of PCP in the 6% fish were about double the values measured in the controls at all time periods. The data for the uptake of TCP are plotted in Figure 4 and for PCP in Figure 5.

Application of ANOVA to the time series data presented in Table 4 determined a significant difference ($P<0.05$) between uptake of TCP in fish exposed to 6% sewage versus controls for the final two sampling periods. However, application of the test to PCP uptake data did not show a significant difference between uptake in controls and uptake in fish exposed to 1%, 6%, or 16% sewage. The ability of rainbow trout to take up PCP directly from the water column has been noted by previous workers (Niimi and Cho 1983).

The reason for the apparent greater TCP uptake in carcasses of fish exposed to 6% sewage than in carcasses of fish exposed to 16% sewage is uncertain, although caution is necessary because the data available was from single pooled samples of individuals rather than replicates. Moreover, the health of the fish during the final two sampling periods may have been a significant factor.

Previous monitoring studies have yielded some information on levels of TCP and PCP in fish from the Lower Fraser River and on Sturgeon Bank. Fish collected in 1973 from 23 stations in the Fraser River between the mouth and Hope (see Albright et al. 1975) were later analysed for chlorophenols under contract to Environment Protection Service (Garrett 1980). These compounds were not present in limited sampling of whole bodies of chinook, sockeye salmon or rainbow trout taken in the estuarine area. Levels of TCP in the range 28.0 to 62.0 ng·g-1 wet weight were reported in staghorn sculpins from the North Arm and PCP levels of 37.5 to 60.0 ng·g-1 were seen in the same samples. Other contaminated organisms included Northern squawfish, lagescale suckers and Dolly Varden char. Recently starry flounders and sediment samples

from the Lower Fraser River and Roberts and Sturgeon Banks were analysed for chlorophenols, PAH's and phthalate esters (Hall et al. 1984). The highest tissue levels of TCP and PCP were observed in single pooled samples of fish caught near the sewage treatment plant outfalls at Iona Island and Annacis Island.

The other pesticides identified in extracts from carcasses of exposed fish were p-p'-DDT, p-p'-DDD and p-p'-DDE. The measured values for these substances are listed in Table 4. Plots of individual components and of Σ DDT against exposure time for different levels of sewage exposure failed to show any consistent trend in the data.

Application of ANOVA to Σ DDT data in Table 4 did not show a significant difference ($P < 0.05$) between uptake and level of exposure to sewage. Mean Σ DDT levels ± S.D. were as follows: Control 100.7 ± 38.4, 1% sewage 96.5 ± 30.2, 6% sewage 96.3 ± 33.8, 16% sewage 105.3 ± 40.3 $ng \cdot g^{-1}$ wet tissue. There are probably two reasons for this finding. The main one is that Iona Island wastewater does not appear to contain a significant burden of any Σ DDT components. We failed to detect the presence of these compounds in the wastewater sampled in both 1982 and 1983 studies. Assuming, however, that low levels of Σ DDT are in fact present, it is probable that Σ DDT is mainly bioaccumulated via the foodchain rather than by direct uptake from the water column. In support of this we cite the work of O'Connor (1984) on the uptake of PCBs (Arochlor 1254) by striped bass. He concluded, based on pharmacokinetic data, that food was the major source of PCB uptake. The DDT family of compounds are about as lipophilic as the PCB's and other workers have shown a linear relationship between Σ DDT and PCB content in fish tissues (Parejko et al. 1975; Norstrom et al. 1978; Bjerk and Brevik, 1980; Gossett et al. 1983a).

Earlier studies on pesticide levels in various fish species from the Lower Fraser River identified p-p-'DDE levels more commonly than either p-p'-DDT or p-p-'DDD (Albright et al. 1975). Sockeye salmon collected in 1973 contained no detectable levels of Σ DDT whereas chinook salmon values ranged from non-detectable to 15.5 $ng \cdot g^{-1}$ wet weight. Rainbow and cutthroat trout showed similarily low levels, whereas a carp from the industrially polluted North Arm contained 1739.9 $ng \cdot g^{-1}$ DDT - the highest uptake measured in the study.

Attempts were made to quantify chlorinated compounds in brain, bile and liver samples from salmon exposed to three concentrations of sewage. Although PCP and TCP residues were identified in all samples, the amounts of tissue or bile available were too small to permit confident quantification.

Five unknown peaks were observed in the pesticide fractions of sewage-exposed fish. These had retention times that fell between pentachloroanisole and DDE and were absent from extracts of control fish. It was assumed that these were most likely organochlorines, although other classes of organics are also detectable by electron capture. The retention times of the five unknown peaks did not match those of any common chlorinated hydrocarbon pesticides and their identities remain unknown.

Lipid extracts of carcasses of salmon, exposed to 1%, 6% and 16% concentrations of Iona Island wastewater, gave yields varying in the range 2.25 to 3.70% with the highest values being recorded in the last two sampling periods (see Table 4). The data available from single pooled samples of fish at each exposure interval was insufficient to justify an ANOVA test. Moreover, the fish were unhealthy near the end of the exposure period and that would be expected to have an effect on lipid yields.

Toxic Substances in Sewage

The toxicity of PCP and TCP to aquatic fauna is well known and has already been discussed. Also it is well understood that PCB's and the DDT family of compounds have similar octanol-water partition functions (Log P values 6.11 for Arochlor 1254, 5.69 for DDD and DDE, 6.19 for DDT; Gossett et al. 1983a). This propensity leads to similar bioaccumulative properties for these materials as already noted. There is evidence that marine fishes affected by fin erosion disease contain higher levels of PCB's in muscle, liver and brain tissues than healthy fish. This correlation was found to apply in fish from contaminated areas in Southern California, Puget Sound, and New York (Sherwood 1982) in which no consistent pattern of heavy metal concentrations was observed. Liver abnormalities were frequently noted in fish suffering from fin erosion disease, especially in Puget Sound and Southern California. These changes in livers included increases in size and (or) lipid content and were thought to relate to DDT and (or) PCB exposure.

Other toxic substances identified in Table 2 include naphthalene and isomers of its mono-, di- and trimethyl analogs. The uptake and biliary excretion of these low molecular-weight PAH's has been discussed (Lech and Melancon 1980). An analytical method for the recovery and quantification of these compounds from clam and oyster tissue samples has been described (Warner 1976). Uptake and release of methylnaphthalenes by oysters, clams, gulf killifish and brown shrimp exposed to No. 2 fuel oil showed bioaccumulation factors that increased with numbers of alkyl groups (Neff et al. 1976). Release rates were species-dependent. Shrimp and fish can metabolize aromatic hydrocarbons and release them more rapidly than do clams and oysters.

An important recent paper summarizes the results of a four-year study on environmental problems associated with chemical contamination of sediments in Puget Sound, Washington, USA (Malins et al. 1984). After analyzing sediment and biota samples from nineteen subareas in Puget Sound for a list of 25 aromatic hydrocarbons, 26 chlorinated organic compounds and 37 metals and other elements, the data were correlated with the incidence of serious liver disorders of three species of demersal fishes using various statistical methods. Strong positive correlations were shown between total aromatic hydrocarbon levels in sediments and prevalence of histopathological conditions in English sole. Other environmental factors showing positive correlations with disease were total heavy metals in English sole and total aromatic hydrocarbons in Pacific staghorn sculpins. The highest concentrations of total aromatic hydrocarbons measured by Malins et al. occurred in sediments from two stations adjoining outfalls discharging municipal, industrial, and storm wastewater. One of these discharges was from the West Point Sewage Treatment Plant, mentioned earlier. Ten of the aromatic

hydrocarbons found in the Puget Sound study are listed in Table 2 of our data from Iona Island wastewater analyses.

Other substances in Table 2 with known or presumed toxic effects on aquatic life include xylenes and alkylbenzenes, dichlorobenzene, phenol, cresols, xylenols and phthalates. The compound 2-phenoxyethanol has anaesthetic properties to fish but was not used in the field work in support of the 1982 Iona Island study. None of the acidic sewage components listed in Table 3 are known to have toxic action on aquatic life.

The United States Environmental Protection Agency has studied the fate of toxic substances in pilot plant treatment facilities representing various practicable levels of municipal wastewater treatment (Petrasek et al., 1983). It was found that 51% of influent levels of Arochlors were transferred to the primary sludge. The corresponding values for phenol, 2,4-xylenol and pentachlorophenol were 4.4%, 0 and 26.0%. Other compounds studied included naphthalene (22.0%), dibutylphthalate (39.0%) and bis-(2-ethylhexyl)-phthalate (63.0%). It is clear from the EPA results that primary treatment of municipal wastewater at Iona Island Sewage Treatment Plant cannot be expected to be an effective barrier, preventing the discharge to the receiving environment of a significant proportion of some of the toxic substances identified in this study.

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Table 1. Recovery of spiking standards from water.

| Number | Compound | R.T. min. | Added $\mu g \cdot L^{-1}$ | Mean Recovery % (n=2) |
|---|---|---|---|---|
| | | Base/Neutrals | | |
| 1 | Phenol | 3.1 | 135 | 48 |
| 2 | Butylbenzene | 4.0 | 102 | 66 |
| 3 | Naphthalene | 5.9 | 117 | 91 |
| 4 | α-Terpineol | 6.2 | 118 | 102 |
| 5 | n-Dodecane | 6.9 | 101 | 111 |
| 6 | n-Tridecane | 7.9 | 107 | 94 |
| 7 | Nicotine | 8.8 | 172 | 72 |
| 8 | n-Tetradecane | 9.6 | 109 | 91 |
| 9 | n-Hexadecane | 12.7 | 112 | 93 |
| I.S. | Dibromodimethoxybenzene | 14.3 | 130 | - |
| 10 | Caffeine | 16.3 | 108 | 153 |
| I.S. | Methyl margarate | 18.5 | 106 | 86 |
| | | Acids | | |
| 11 | Benzoic | 4.3 | 103 | 104 |
| 12 | Phenylpropionic | 7.5 | 101 | 101 |
| 13 | Phenylbutyric | 8.7 | 101 | 100 |
| I.S.* | Dibromodimethoxybenzene | 14.3 | 130 | 110 |
| I.S. | Methyl margarate | 18.5 | 106 | - |

* Internal Standard

Table 2. Compounds identified in base/neutral fractions of raw and treated Iona Island wastewater samples+

| Compound | Concentration (n = 6) $\mu g \cdot L^{-1}$ | Compound | Concentration (n = 6) $\mu g \cdot L^{-1}$ |
|---|---|---|---|
| Xylene | 12-110 | methyl naphthalene | 0-10 |
| Xylene | 7-59 | $C_{13}$-hydrocarbon** | 7-63 |
| Cyclohex-2-enone | | methyl naphthalene | |
| trichloropropene | | nicotine** | 5-29 |
| $C_3$-alkylbenzene | | $C_{14}$-hydrocarbon** | 2-58 |
| $C_3$-alkylbenzene | 2-123 | dimethyl naphthalene | 0-11 |
| $C_3$-alkylbenzene | | dimethyl naphthalene | |
| phenol** | | dimethyl phthalate | |
| $C_{10}$-hydrocarbon | | $C_{15}$-hydrocarbon | 3-59 |
| dichlorobenzene | 0-2 | trimethyl naphthalene | |
| n-butylbenzene** | | diethyl phthalate | |
| dichlorobenzene | | $C_{16}$-hydrocarbon** | 6-61 |
| benzyl alcohol | 26-64 | tributyl phosphate | |
| $C_4$-alkylbenzene | | dibromodimethoxybenzene* | 200 |
| cresol | 7-29 | | |
| $C_4$-alkylbenzene | | $C_{18}$-hydrocarbon | |
| $C_4$-alkylbenzene | | caffeine** | 2-41 |
| cresol | | $C_{19}$-hydrocarbon | |
| $C_{11}$-hydrocarbon | | 2-benzyl-4-chlorophenol | |
| cresol | | dibutyl phthalate | 15-52 |
| phenylethanol | | methyl margarate* | 200 |
| xylenol | 174 | $C_{21}$-hydrocarbon | |
| xylenol | | $C_{23}$-hydrocarbon | |
| $C_4$-alkylbenzene | | $C_{24}$-hydrocarbon | |
| camphor | | $C_{25}$-hydrocarbon | |
| $C_4$-alkylbenzene | | di-2-ethylhexylphthalate | 0-49 |
| isoborneol | | $C_{26}$-hydrocarbon | |
| menthol | 5-29 | $C_{27}$-hydrocarbon | |
| naphthalene** | | $C_{28}$-hydrocarbon | |
| $\alpha$-terpineol** | 21-191 | Coprostanol | |
| $C_{12}$-hydrocarbon** | | Cholesterol | |
| 2-phenoxyethanol | | | |
| methyl ethyl phenol | | | |
| carvone | 10-16 | | |

* internal standard

** calibration standard

+ tentative identifications based on electron impact mass spectra and matching of spectra and retention times with standards, where available. Gas chromatographic separation on fused silica Durabond - 5 capillary column (30 m x 0.25 mm). Compounds arranged in order of elution. Repetition of compound names indicates the presence of structural isomers.

Table 3. Acids identified as methyl esters in acidic fractions of raw and treated Iona Island wastewater samples+

| Compound | Structure | Concentration range (n = 6) $\mu g \cdot L^{-1}$ |
|---|---|---|
| isovaleric | | |
| Caproic | $C_6:0$ | |
| caproic | $C_6:0$ | |
| malic | | |
| 2-furancarboxylic | | |
| succinic | | |
| cyclohexane-carboxylic | | |
| benzoic** | | 11-194 |
| caprylic | $C_8:0$ | 27-446 |
| glutaric | | |
| phenylacetic | | 32-344 |
| salicylic** | | |
| pelargonic | $C_9:0$ | |
| crotonic | | |
| phenylpropionic** | | 7-79 |
| 3-methyladipic | | |
| hydroxyphenyl-propionic | | |
| capric | $C_{10}:0$ | 21-243 |
| pimelic | | |
| cinnamic | | |
| phthalic | | |
| lauric | $C_{12}:0$ | 49-354 |
| azelaic | | |
| dibromodimethoxy-benzene* | | |
| myristic | $C_{14}:0$ | 84-543 |
| pentadecylic | $C_{15}:0$ | 19-36 |
| pentadecylic | $C_{15}:0$ | 29-88 |
| palmitoleic | $C_{16}:1$ | 15-188 |
| palmitic | $C_{16}:0$ | 530-1800 |
| margaric | $C_{17}:0$ | |
| margaric* | $C_{17}:0$ | 0-32 |
| linoleic | $C_{18}:2$ | 0-163 |
| oleic | $C_{18}:1$ | 23-651 |
| elaidic | $C_{18}:1$ | 77-233 |
| stearic | $C_{18}:0$ | 223-584 |
| eicosenoic | $C_{20}:1$ | 2-178 |

Table 3 (cont'd)

| Compound | Structure | Concentration range (n = 6) $\mu g \cdot L^{-1}$ |
|---|---|---|
| arachidic | $C_{20}$:0 | |
| behenic | $C_{22}$:0 | 15-41 |
| lignoceric | $C_{24}$:0 | |

* internal standard

** calibration standard

\+ tentative identifications based on electron impact mass spectra and comparison of retention times with standards, where available. Gas chromatographic separation on fused silica Durabond - 5 capillary column (30 m x 0.25 mm). Compounds arranged in order of elution. Repetition of compound names indicates presence of structural isomers.

Table 4. Levels of chlorinated phenols and chlorinated hydrocarbons in carcasses of chinook salmon exposed to three concentrations of primary treated sewage.

| $ng \cdot g^{-1}$ wet tissue* | | | | | | | Days of | | $ng \cdot g^{-1}$ lipid* | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TCP | PCP | DDE | DDD | DDT | ΣDDT | PCB | exposure | Lipid % | TCP | PCP | DDE | DDD | DDT | ΣDDT | PCB |
| | | | | | | | Control 0% sewage | | | | | | | | |
| 12 | 10 | 64 | 60 | 48 | 172 | 97 | -1 | 2.74 | 442 | 379 | 2331 | 2206 | 1769 | 6306 | 1778 |
| 7 | 12 | 50 | 22 | 21 | 93 | 41 | 3 | 2.82 | 257 | 420 | 1769 | 785 | 758 | 3312 | 1439 |
| 5 | 5 | 40 | 14 | 17 | 71 | NQ | 6 | 2.24 | 213 | 222 | 1766 | 624 | 779 | 3169 | NQ |
| 9 | 7 | 48 | 14 | 10 | 72 | NQ | 9 | 2.56 | 334 | 284 | 1861 | 550 | 384 | 2795 | NQ |
| 7 | 5 | 46 | 21 | 15 | 82 | NQ | 13 | 3.66 | 196 | 147 | 1217 | 569 | 400 | 2186 | NQ |
| 58 | 23 | 52 | 52 | 10 | 114 | 70 | 19 | 2.61 | 2211 | 868 | 1985 | 1985 | 364 | 4334 | 1336 |
| | | | | | | | 1% sewage | | | | | | | | |
| 40 | 16 | 87 | 26 | 11 | 124 | 19 | (-1) | 2.59 | 1530 | 606 | 3386 | 995 | 436 | 4817 | 718 |
| 8 | 6 | 51 | 21 | 11 | 83 | 79 | 3 | 2.83 | 272 | 206 | 1798 | 754 | 392 | 2944 | 2810 |
| 8 | 6 | 44 | 23 | 8 | 75 | 55 | 6 | 2.74 | 275 | 207 | 1622 | 846 | 281 | 2749 | 2020 |
| 10 | 6 | 50 | 28 | 10 | 88 | 54 | 9 | 2.58 | 369 | 247 | 1955 | 1072 | 407 | 3434 | 2080 |
| 19 | 14 | 65 | 64 | 14 | 143 | 76 | 13 | 3.28 | 566 | 433 | 1989 | 1964 | 418 | 4371 | 2330 |
| 26 | 5 | 42 | 15 | 9 | 66 | 36 | 19 | 3.60 | 721 | 145 | 1154 | 423 | 246 | 1823 | 1020 |
| | | | | | | | 6% sewage | | | | | | | | |
| 77 | 21 | 73 | 21 | 8 | 102 | 28 | (-1) | 3.07 | 2530 | 673 | 2400 | 706 | 265 | 3371 | 910 |
| 74 | 22 | 65 | 26 | 13 | 104 | 44 | 3 | 2.78 | 2676 | 792 | 2332 | 928 | 480 | 3740 | 792 |
| 75 | 14 | 34 | 11 | 7 | 52 | 79 | 6 | 2.96 | 2528 | 482 | 1137 | 382 | 227 | 1746 | 1344 |
| 103 | 16 | 40 | 13 | 7 | 60 | 71 | 9 | 3.21 | 3214 | 500 | 1232 | 398 | 211 | 1841 | 1104 |
| 165 | 28 | 83 | 28 | 13 | 124 | 108 | 13 | 3.39 | 4888 | 834 | 2445 | 842 | 383 | 3670 | 1606 |
| 247 | 50 | 84 | 31 | 21 | 136 | 104 | 19 | 3.56 | 6960 | 1397 | 2358 | 886 | 602 | 3846 | 1466 |

Table 4 (cont'd)

| $ng \cdot g^{-1}$ wet tissue* | | | | | | | Days of | | $ng \cdot g^{-1}$ lipid* | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TCP | PCP | DDE | DDD | DDT | ΣDDT | PCB | exposure | Lipid % | TCP | PCP | DDE | DDD | DDT | ΣDDT | PCB |
| | | | | | | | 16% sewage | | | | | | | | |
| 10 | 17 | 73 | 46 | 20 | 139 | 83 | -1 | 3.01 | 320 | 550 | 2431 | 1522 | 666 | 4619 | 1380 |
| 9 | 16 | 37 | 12 | 7 | 56 | 87 | 3 | 2.81 | 331 | 577 | 1330 | 421 | 255 | 2006 | 1551 |
| 11 | 22 | 51 | 26 | 15 | 92 | 91 | 6 | 2.65 | 402 | 840 | 1929 | 932 | 582 | 3443 | 1724 |
| 9 | 15 | 41 | 14 | 12 | 67 | 72 | 9 | 2.73 | 345 | 543 | 1517 | 506 | 451 | 2474 | 1319 |
| 145 | 61 | 88 | 21 | 12 | 121 | 110 | 13 | 3.66 | 4895 | 2049 | 2980 | 695 | 420 | 4095 | 1862 |
| 132 | 62 | 74 | 85 | 18 | 157 | 72 | 19 | 3.41 | 3862 | 1808 | 2163 | 1916 | 522 | 4601 | 1056 |

NQ = not quantifiable, amount below limit of confident quantification estimated as 10 $ng \cdot g^{-1}$ wet weight or 300 $ng \cdot g^{-1}$ of lipid for Arochlor 1254, based on 3% lipid content.

*data not corrected for efficiency of recovery. Pooled samples of individuals without replicates.

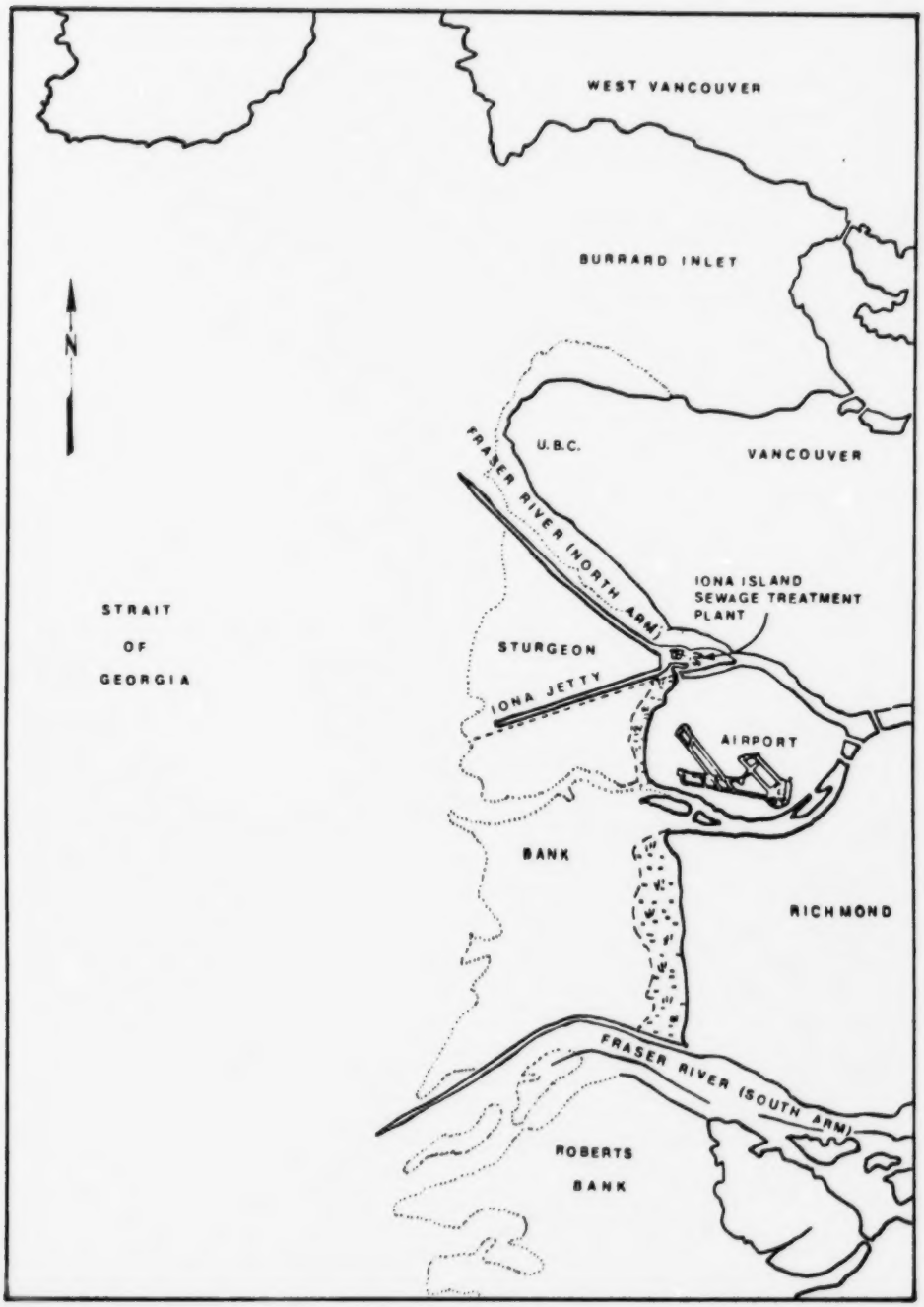


Fig. 1. Location of Iona Island Sewage Treatment Plant on the Fraser River estuary.

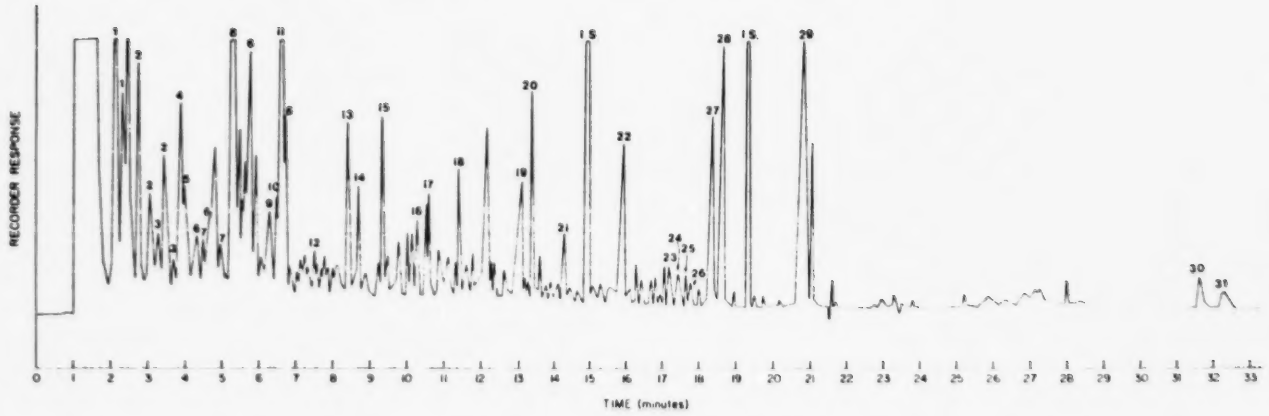


Fig. 2. Gas chromatogram of a base/neutrals fraction of a grab sample of Iona Island raw sewage (Sept. 15, 1982). Internal standards 1,2-dibromo-4,5-dimethoxybenzene followed by methyl margarate. Peaks were identified by GC-MS analysis as follows: 1 xylenes, 2 $C_3$-alkylbenzenes, 3 dichlorobenzenes, 4 benzyl alcohol, 5 butylbenzene, 6 $C_4$-alkylbenzenes, 7 cresols, 8 xylenol, 9 menthol, 10 naphthalene, 11 α-terpineol, 12 carvone, 13 $C_{13}$-paraffin, 14 methyl naphthalene, 15 nicotine, 16 $C_{14}$ paraffin, 17 dimethyl naphthalene, 18 $C_{15}$-paraffin, 19 free lauric acid, 20 diethyl phthalate, 21 tributyl phosphate, 22 free myristic acid, 23 caffeine, 24 $C_{19}$-paraffin, 25 unknown monochloro compound MW302, 26 2-benzyl-4-chlorophenol, 27 free palmitoleic acid, 28 free palmitic acid, 29 free stearic acid, 30 coprostanol, 31 cholesterol.

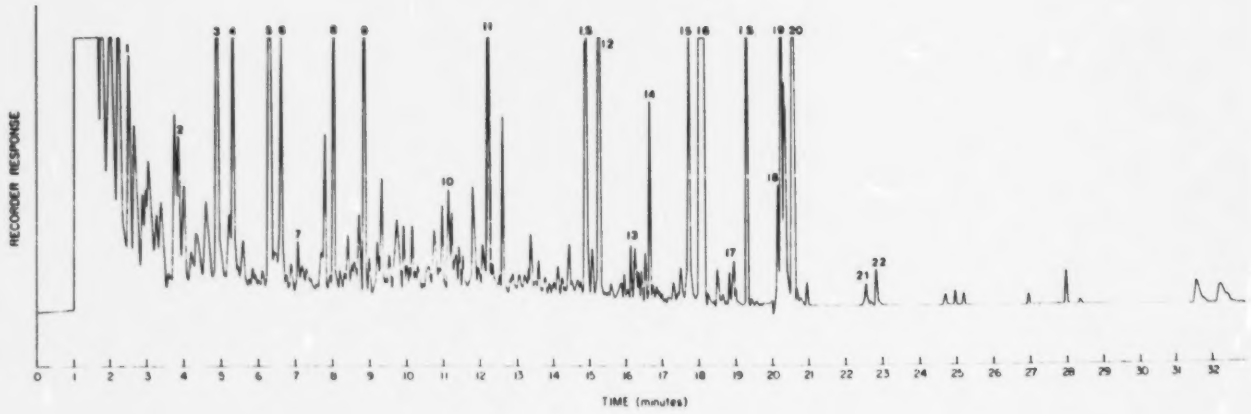


Fig. 3. Gas chromatogram of an acid fraction of a grab sample of Iona Island raw sewage (Sept. 15, 1982). Internal standards 1, 2-dibromo-4,5-dimethoxybenzene followed by methyl margarate. Peaks were identified by GC-MS analysis as follows: 1 caproic, 2 succinic, 3 benzoic, 4 caprylic, 5 phenylacetic, 6 salicylic, 7 pelargonic, 8 phenylpropionic, 9 capric, 10 phthalic, 11 lauric, 12 myristic, 13 branched chain pentadecanoics, 14 pentadecanoic, 15 palmitoleic, 16 palmitic, 17 branched chain heptadecanoics, 18 linoleic, 19 oleic and isomers, 20 stearic, 21 erucic and 22 behenic acids.

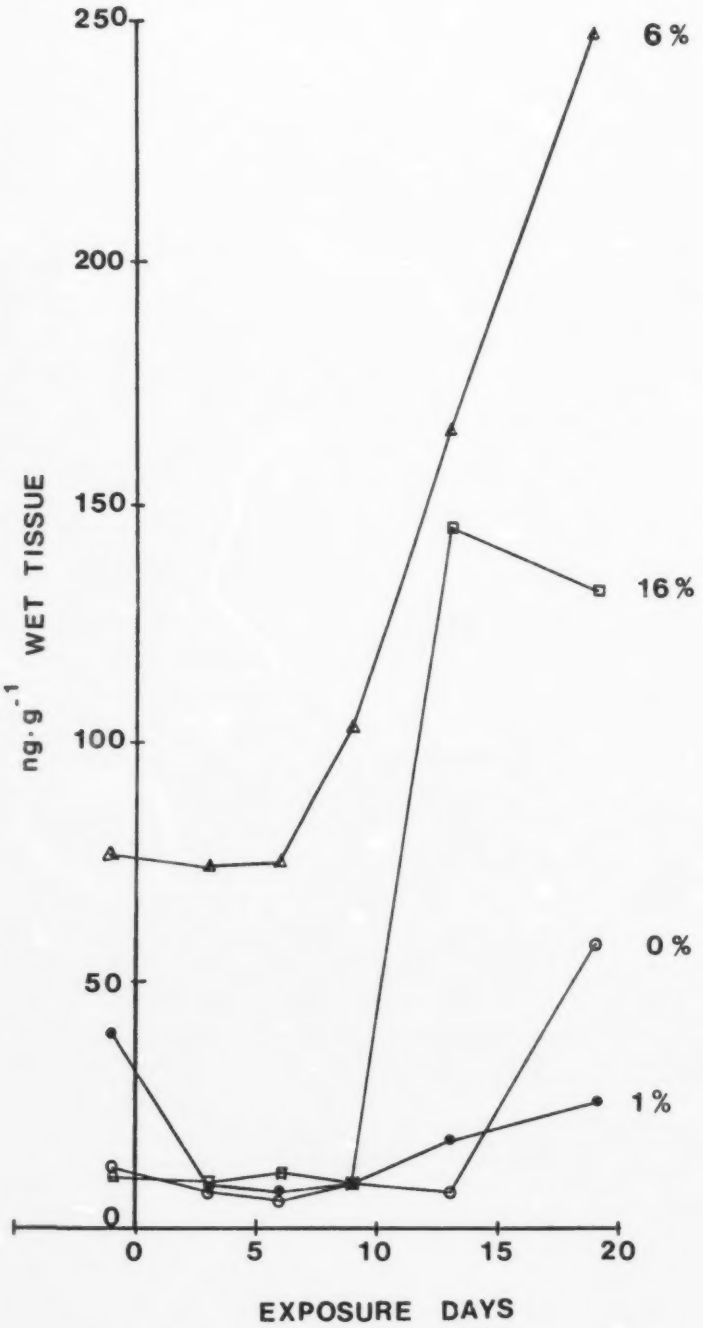


Fig. 4. Uptake of tetrachlorophenol (TCP) in the carcasses of juvenile chinook salmon exposed to 0%, 1%, 6%, and 16% concentrations of Iona Island Sewage Treatment Plant wastewater, June-July 1982. Diluent water was taken from the Fraser River, North Arm.

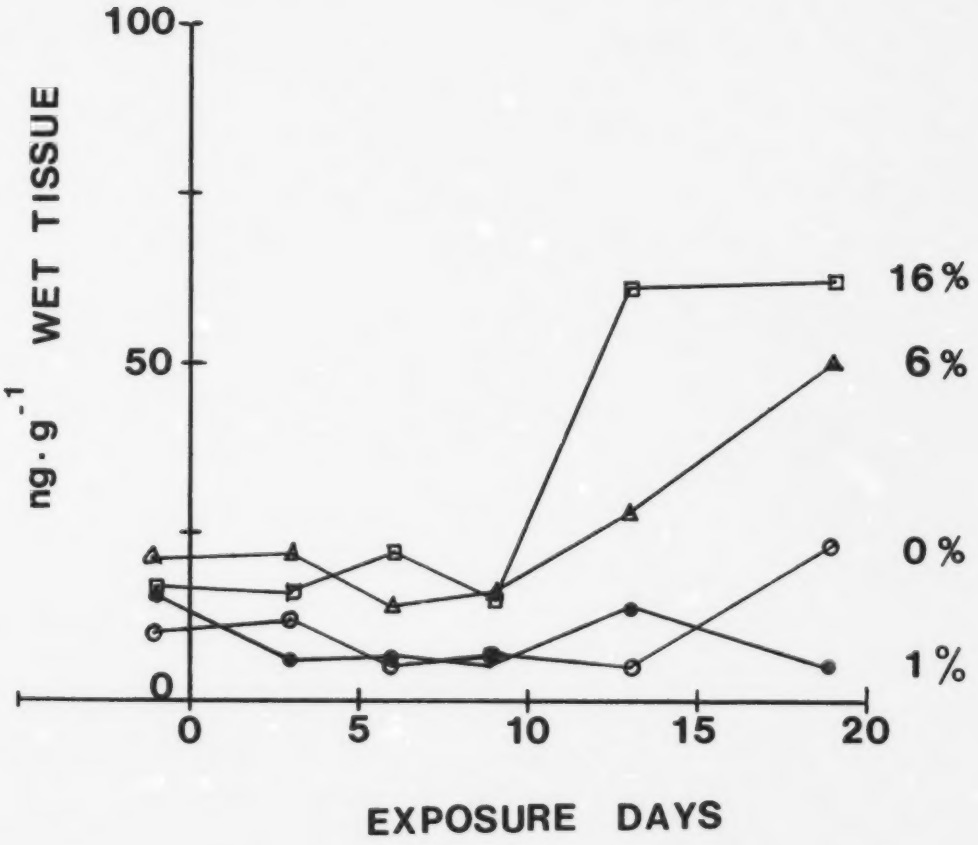


Fig. 5. Uptake of pentachlorophenol (PCP) in the carcasses of juvenile chinook salmon exposed to 0%, 1%, 6%, and 16% concentrations of Iona Island Sewage Treatment Plant wastewater, June-July 1982. Diluent water was taken from the Fraser River, North Arm.